Comparing Testing Methods for Stainless Steel Corrosion Resistance

Testing the corrosion resistance of stainless steel fluid components employed in ultrahigh purity (UHP) distribution systems is a critical process to ensure the components meet industry-specified standards. To handle the corrosive gases used in UHP service, such components feature a passive chromium-enriched oxide layer on their wetted surfaces. Suppliers use two common techniques – surface chemistry analysis and critical pitting temperature (CPT) – to evaluate the passive layer's ability to resist corrosion.

A fundamental difference between the tests is the total surface area that is subject to testing. Surface chemistry analyzes a series of discrete points on a sample, representing just a small percentage of the total surface area that would be exposed to corrosive gases in service. CPT testing analyzes the full wetted surface area.

Both CPT and surface chemistry tests have their place in industry. However, the CPT test shows promise for wider adoption as it can provide more consistent results, in less time, and with a lower cost for select metals. This article focuses on the key differences between the two methods, including testing methodologies, logistics, and limitations.

Enhancing Corrosion Resistance

It is important to first understand how component suppliers enhance the corrosion resistance of stainless steel.

Stainless steel is the industry-preferred material for UHP products used in corrosive gas delivery systems due to the metal's inherent resistance to corrosion through an auto-passivating surface. To enhance this property, the base metal requires further processing in the form of electropolishing and passivation.

Electropolishing creates a chromium-enriched oxide layer on the metal to enhance its corrosion resistance. During the process, surface iron is removed from the component and electroplated to a cathode. The component's surface is typically smoothed during this process, improving surface finish and reducing the overall wetted area.

Passivation dissolves free iron on a component's surface and removes electrolytes that were not removed by post-electropolishing rinsing. The resulting chromium oxide layer is less electrochemically reactive and is, therefore, more resistant to corrosion.

Both electropolishing and passivation increase the chrome to iron (Cr/Fe) ratio and chrome oxide to iron oxide (CrO/FeO) ratio on the metal's surface. Typical 316L stainless steel electropolished specifications require a Cr/Fe ratio of 1.5 to 2.0, improved from 0.5 or less in untreated material. It is important to note that higher Cr/Fe ratios are possible but are often accompanied by a thicker oxide layer which may actually degrade a material's corrosion resistance due to surface porosity.

Testing Methodologies

Surface Chemistry Analysis. Traditional surface chemistry analysis techniques confirm the thickness of the metal's passivated oxide surface layer as well as the surface Cr/Fe and CrO/FeO ratios. Various points on the metal's wetted surface are isolated and subjected to analysis. Based on the test findings, the metal is assumed to have a certain level of corrosion resistance.

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Surface chemistry analysis methods are specified in SEMI F60 [1] and SEMI F72 [2]. Common methods are Auger Electron Spectroscopy (AES), Electron Spectroscopy for Chemical Analysis (ESCA), and Secondary Ion Mass Spectroscopy (SIMS), each of which uses a different technique to analyze discrete locations on a sample. For example, in AES testing, a high-energy electron beam is directed at the sample causing electrons to be released. The emitted electrons are analyzed to determine the atomic concentration of selected elements, such as iron, chromium, and oxygen.

In some cases, surface chemistry analysis will show an acceptable surface chemistry in one location on the sample but unacceptable chemistry at another location nearby. Thus, test results may vary since the examination occurs at very discrete points rather than over the full surface.

CPT Testing. CPT testing evaluates the entire passivated surface of the sample. The sample's chromium oxide surface layer is stressed to the point of failure to determine its resistance to localized pitting corrosion. The temperature at which the surface layer fails is known as the CPT.

The CPT test method applies an electrical potential across the passive layer of the component through a conductive liquid. The quality of the passive layer is evaluated by increasing the test temperature to determine the component's dielectric strength. Comparing CPTs from multiple samples indicates their relative dielectric strengths, and thereby provides an indication of the quality of the passive layers and corrosion resistance. Typical industry specifications require the CPT for 316 stainless steel to be greater than 13ºC.

Figure 2

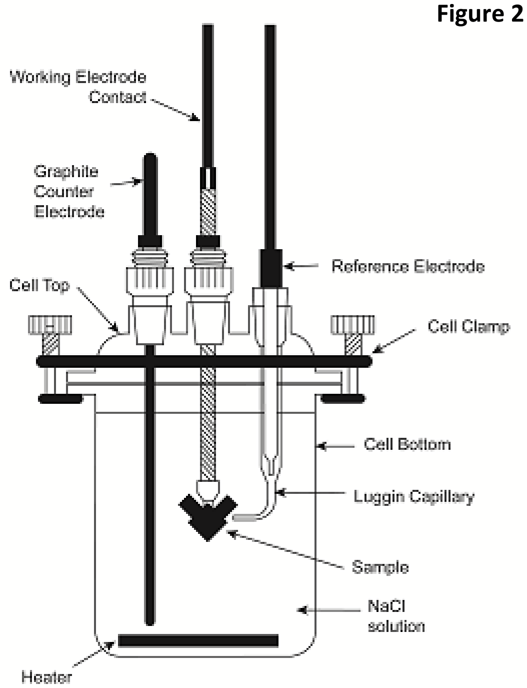

Figure 1 shows a common set-up for CPT testing. A sample is attached to an electrode lead and immersed in a heated electrolyte bath. The bath temperature is increased gradually and evenly throughout the chamber. Operators maintain a constant electrical potential and monitor the current passing through the wetted portion of the sample to determine when an established current limit is achieved. At this point, the electrical potential breaks down the passive layer.

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Figure 2

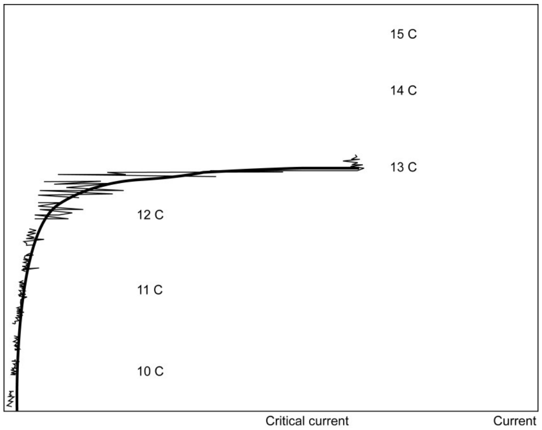

Figure 2 plots the temperature and current flow of a CPT test. A spike in the current passing through the metal occurred at 13ºC. At this point, the weakest portion of the passive layer broke free from the sample, forming a pit (see Figure 3) that exposed the higher iron-content material below and allowed current to flow freely. Therefore, the CPT for this sample is 13ºC.

CPT testing is based on ASTM G150 [3]. SEMI's F077 [4] is an adaptation of ASTM G150 and is related specifically to semiconductor systems.

Figure 3a

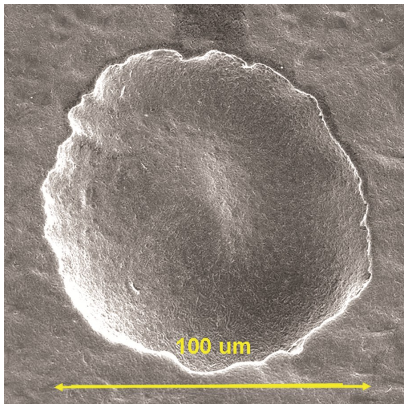

Figure 3b

Testing Logistics

CPT testing offers potential time and cost savings compared to surface chemistry testing based simply on the logistics for running these tests.

Surface chemistry analysis often requires the use of one of a limited number of qualified third-party labs. Therefore, one must ship samples to the lab and wait for results. The wait may be lengthy, depending on the lab's workload and capacity. Companies have the option to set up surface chemistry analysis equipment in-house, but the equipment is costly and the training is rigorous.

The CPT testing set-up is relatively simple, and companies can create a testing apparatus for a modest investment. Alternatively, local labs may have open capacity for CPT testing. Whether testing is performed in-house or locally, CPT test results may be available in hours or days compared to weeks for surface chemistry results.

Testing Limitations

Each testing method has its limitations, which factor into which test is most viable for a particular sample.

Surface Area Availability. Only the passivated portions of a sample need to be tested for corrosion resistance. For example, the interior of a UHP tube butt weld fitting may be the only area with a passive oxide layer.

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For surface chemistry analysis, it is best to analyze a relatively flat surface on the sample. The curved interior of the UHP tube fitting may, therefore, not be conducive to accurate surface chemistry analysis.

When the available surface is curved, CPT testing may be preferred. Remember that CPT testing involves full immersion of the sample. Therefore, the non-passivated areas of the component need to be masked with an insulating paint (see Figure 4).

Repeatability. CPT testing is highly repeatable as it consistently finds the weakest point on the entire passivated surface layer of samples. By contrast, surface chemistry analysis may find the weakest or strongest point, or some point in between. Each spot may have a different chromium oxide thickness, so a sample could pass a test at its best point even if a majority of the surface area is sub par.

Material Compatibility. Surface chemistry analysis can be conducted on nearly all metals. CPT testing, however, is limited to materials that are prone to pitting corrosion. Stainless steel and some related alloys qualify due to their pitting corrosion susceptibility.

Detection Limits. Further, the temperature extreme of a CPT test is limited to the boiling point of the bath. If the solution boils before the sample fails, then the CPT is unknown and the test is inconclusive. A CPT greater than 13ºC is typically recommended for 316 stainless steel.

Figure 4

Conclusion

Both surface chemistry and CPT testing present the industry with options for verifying that stainless steel components meet corrosion resistance standards for UHP gas service. Yet each test has its limitations. Therefore, the preferred test is at the discretion of the user based on material compatibility and availability of testing resources. Under the right set of conditions and for appropriate materials, the CPT test may be quicker, more economical, and return more consistent results than a surface chemistry test.

Remember that the success rate for passing either CPT or surface chemistry tests originates with a component supplier's adherence to sound process controls that ensure quality throughout the design, manufacturing, and delivery processes.

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